(1) That four to six drops of a carefully neutralized, so-called 1 per cent alcoholic solution of phenolphthalein be used as indicator, and a "blank" determination on water and indicator be used to correct the titration.

(2) That the weight taken for titration of honeys grading "Amber" or darker should not exceed 5 to 6 grams.

(3) That the charge of honey should be entirely dissolved in 75 ml. or more of CO_2-free water before starting the titration.

(4) That the recorded end point of the titration should fulfill the following requirements: (a) the pinkish coloration due to the indicator should persist for at least 10 seconds; and (b) mixing should be adequate to produce a homogeneous solution.

(5) That titration to *p*H 8.30 (corrected) measured by *p*H meter shall be considered equivalent to titration using phenolphthalein indicator.

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REPORT ON HONEY*

By GEORGE P. WALTON (Eastern Regional Research Laboratory[1] Philadelphia 18, Pennsylvania), *Associate Referee*

After the 1948 meeting, Committee D recommended further study of the official method for determination of free acid in honey, 34.99, to establish the end point more accurately; also a study of methods for detection of adulterants in honey, particularly commercial sirups.

A brief survey to aid in planning work for the second study indicated that the primary objective should be collaborative testing of two methods developed in the Federal Food and Drug Administration, one by W. O. Winkler, Washington, D. C., the other by George McClellan, Houston, Texas. Both are methods for detecting adulteration of honey with invert sugar sirup; neither one has been published. Since the time available for the work was limited, and details of the methods were not readily available to persons outside of the Food and Drug Administration, collaborative work was not attempted. However, a heavy invert sugar sirup was carefully prepared from pure sucrose and invertase with added organic acids to simulate the composition of honey, and this and authentic samples of natural honeys will be available to next year's Referee for collaborative work.[2]

Determination of the free acid in light-colored honeys can be accomplished satisfactorily by the present official method; but concordant titration values are less likely to be obtained with increasing depth of color in the honeys. For example, three lots of honey ranging in color grades from "white" to dark were each carefully mixed and subsampled for collaborative work. Table 1 gives the titration results obtained by Patricia J. Dougherty, the one collaborator reporting, and this Referee.

It is apparent that a charge of 10 grams is too large for dark honeys.

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* Report of a study made under the Research and Marketing Act of 1946.

[1] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture.

[2] Because of the limited time available for A.O.A.C. work, the writer has requested that he not be reappointed Associate Referee.

TABLE 1.—*Titration of A.O.A.C. Samples*

(At room temperature; after 10 seconds of phenolphthalein "pink" coloration)

SAMPLE NO.	COLOR	COLLABORATOR		
		A	B	B (BY *p*H METER)
		ML 0.1 *N* NaOH REQUIRED PER 100 G HONEY AT—		
		20°C.	*27°C.*	*25°C.*
A.O.A.C. 1	White	16.62	16.65	16.80
A.O.A.C. 2	Dark	31.05	50.15[1]	49.95
A.O.A.C. 3	White	22.11	21.89	21.75

[1] Determined on one-half usual weight of honey.

The dark color of the solution titrated masks the change in the color of the phenolphthalein indicator.

Some advantage was noted by conducting the titration according to the official method for determining the acidity of wines **15.22(a),**

In order to determine the true titration end points for the three honeys, the progress of the neutralization was followed with a *p*H meter. The values in the last column of Table 1, correspond to *p*H 8.3. Evidently the pink tint of phenolphthalein was discernible in the white honey at a slightly lower *p*H than 8.3.

At *p*H 7.0, the respective titration figures were 10.7, 35.5, and approximately 16 ml. of 0.1 *N* alkali. Evidently the constituents of honey exert the equivalent of a strong buffer effect on alkali added after the *p*H 7 point has been passed.

Titrating at 37°–38°C., instead of at 20°, or at 27°, gave significantly higher results only for sample 1. The increase ranged from 0.16 to 2.35 ml.

When the titrations at room temperature were continued until the pink coloration due to the indicator, or a *p*H of 8.3, had persisted for 30 seconds and again for 90 seconds, instead of approximately 10 seconds, the interval for the recorded titration, increases were recorded in every case. Collaborator A used an average of 1.36 ml additional alkali for 30 seconds' persistence of the pink color and further additional 1.25 ml for 90 seconds. Maintaining the *p*H at approximately 8.30 (by *p*H meter) for 30 seconds required an average of 1.03 ml more alkali than for the 10-second titration, but only 0.1 to 0.2 ml additional to maintain that state for 90 seconds. Collaborator B's titration with phenolphthalein agreed with that by *p*H meter. At 37°–38°C., the increments of alkali required to maintain the end-point concentration were of about the same magnitude as at 20° to 27°.

RECOMMENDATIONS*

On the basis of this work, in titrating the free acids in honey, it is recommended—

* For report of Subcommittee D and action of the Association, see *This Journal*, 33, 68 (1950).